INDIANA DEPARTMENT OF TRANSPORTATION
MATERIALS AND TESTS DIVISION

QUANTITATIVE EXTRACTION OF ASPHALT/BINDER AND GRADATION OF EXTRACTED AGGREGATE FROM HMA MIXTURES
ITM No. 571-02T

1.0 SCOPE.

1.1 This method of test covers the procedure for the quantitative determination of the asphalt/binder content and gradation of the extracted aggregate of asphalt paving mixtures.

1.2 The HMA mixture is extracted with a suitable solvent, depending on the type of extraction apparatus used. The asphalt/binder content is calculated by determining the difference of the weight (mass) of the HMA mixture and the extracted aggregate, fibers if used, and the fines recovered from the extracted solvent and water rinse if required. The gradation is then determined of the extracted aggregate.

1.3 The values stated in either acceptable English or SI metric units are to be regarded separately as standard, as appropriate for a specification with which this ITM is used. Within the text, SI metric units are shown in parenthesis. The values stated in each system may not be exact equivalents; therefore each system shall be used independently of the other, without combining values in any way.

1.4 This ITM may involve hazardous materials, operations, and equipment. This ITM does not purport to address all of the safety problems associated with the ITMs use. The ITM user's responsibility is to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.

2.0 REFERENCES.

2.1 AASHTO Standards.

M 231	Weighing Devices Used in the Testing of Materials
T 30	Mechanical Analysis of Extracted Aggregate
T 164	Quantative Extraction of Bitumen from Bituminous Paving Mixtures

2.2 ITM Standards.

572	Drying HMA Mixtures
580	Sampling HMA

3.0 TERMINOLOGY.

3.1 Terms and Abbreviations. Definitions for terms and abbreviations shall be in accordance with the Department's Standard Specifications, Section 101.

4.0 SIGNIFICANCE AND USE.

This ITM will be used to determine the asphalt/binder content and gradation of the extracted asphalt paving mixture.

5.0 APPARATUS.

5.1 Oven, capable of maintaining the temperature at 221 ± 9 °F (105 ± 5 °C)

5.2 Electric skillet, with a thermostatic heat control capable of heating to 221 °F (105 °C)

5.3 Spatulas and trowels as needed

5.4 Pans and containers as needed

5.5 Armored thermometer with range of 100 °F (40 °C) to 450 °F (230 °C), readable to 5 °F (2 °C)

5.6 Balance, a Class G2, conforming to the requirements of AASHTO M 231

5.7 Wash bottle

5.8 Stiff bristle brush, 1 in. (25 mm) in diameter

5.9 Sieves conforming to the requirements of AASHTO T 30

6.0 REAGENTS.

6.1 Extraction Solvent

6.2 Trichloroethylene, Reagent Grade or Technical Grade Type 1, Federal Specification O-T-634

7.0 SAFETY PRECAUTIONS.

7.1 The reagent solvents are toxic as shown below:

Reagent	Maximum Acceptable Concentration for 8 h Exposure, ppm
Extraction Solvent	Not Established
Trichloroethylene	100

7.2 Provide adequate ventilation. Avoid inhalation of vapor. The ventilation fan must be operating during the testing.

7.3 The exhaust from the vacuum pump must be vented outside.

7.4 The Trichloroethylene has a boiling point of 189 °F (87 °C). The Material Safety Data Sheet will list the boiling point of the extraction solvent.

7.5 The extraction solvent shall be an approved solvent.

8.0 SAMPLING. Sampling shall conform to the requirements of ITM 580.

9.0 PREPARATION OF SAMPLE.

9.1 If the sample is not sufficiently soft to separate with a spatula or a trowel, place it in a large flat pan and heat to a maximum of 221 ± 9 °F (105 ± 5 °C) only until it can be handled. Separate the sample as uniformly as possible, using care not to fracture the mineral aggregate.

9.2 The approximate minimum size of the sample will be in accordance with the following:

Size of Sample	
Mixture Designation	**Minimum Weight (mass) of Sample, g**
4.75 mm	1000
9.5 mm	1500
12.5 mm	2000
19.0 mm, C19.0 mm	3000
25.0 mm, C25.0 mm	4000
37.5 mm	6000

10.0 PROCEDURES.

10.1 METHOD A - (USING A CENTRIFUGE EXTRACTOR).

10.1.1 METHOD SPECIFIC APPARATUS. In addition to the apparatus listed in 5.0, the following apparatus is required for Method A.

a) Centrifuge Extractor (AASHTO T 164)

b) Filter rings, to fit the rim of the centrifuge bowl

10.1.2 USING A CENTRIFUGE EXTRACTOR.

a) Dry the sample to constant weight (mass) in accordance with ITM 572.

b) Place the sample in a tared extraction bowl and determine the weight (mass).

c) Cover the test sample in the bowl with Trichloroethylene and allow sufficient time for the solvent to break up the sample. The amount of solvent needed will be governed by the size of the centrifuge bowl.

d) Assemble the centrifuge extractor with a dry weighed filter paper in place. Clamp the cover on tightly and place a container under the drain to collect the extracted solvent.

e) Start the centrifuge revolving slowly and gradually increase the speed to a maximum of 3600 rpm., or until the solvent ceases to flow from the drain. Allow the extractor to stop and add solvent as in 10.1.2 c). Repeat the procedure until the extracted solvent is a light straw color.

f) Collect the extracted solvent in a suitable container.

g) Determine the amount of fines in the extracted solvent in accordance with AASHTO T 164 (11.6).

h) Dry the extracted aggregate and filter to a constant weight (mass) in the oven or skillet at 221 ± 9 °F (105 ± 5 °C).

10.1.3 CALCULATION USING CENTRIFUGE.

a) The asphalt/binder content in percent is calculated by the following formula:

$$\text{Asphalt/Binder Content, \%} = \frac{W1 - (W2 + W3)}{W1} \times 100$$

Where:

W1 = weight (mass) of sample, g
W2 = weight (mass) of extracted aggregate, g
W3 = weight (mass) of fines in extracted solvent, g

10.2 METHOD B - (USING A VACUUM EXTRACTOR).

10.2.1 METHOD SPECIFIC APPARATUS. In addition to the apparatus listed in 5.0 the following is required:

a) Vacuum extractor

b) Filter paper, medium grade, fast filtering of the diameter required to fit the extractor, (Eaton-Dikeman #633-70)

c) Vacuum pump

d) Pan, round, bowl type, stainless steel

e) No. 200 (75 µm) sieve

10.2.2 USING A VACUUM EXTRACTOR NO FIBERS.

a) Weigh approximately 50 g (record exact weight (mass)) of a filtering aid, such as celite, into a 1000 mL flask, add 500 mL of extraction solvent, and swirl until the filtering aid is completely in suspension. 100 g of filtering aid may be used if the solvent does not readily pass through the filter. Immediately pour the solution onto the filter. Start the vacuum pump and let it run until the pad formed by the filtering aid is surface dry and begins to crack slightly. Collect the solvent which goes through the filter in a flask, and pour it onto the filter.

b) Dry the sample to a constant weight (mass) in accordance with ITM 572. Determine the weight (mass) of a dry filter paper at 221 ± 9 °F (105 ± 5 °C).

c) If the sample is in an oven bag, remove the sample from the bag, and weigh the sample. Add enough solvent to cover the sample and stir vigorously. (Soaking the sample after stirring for several minutes may be beneficial in removing the asphalt/binder from the aggregate). Stirring should continue until the sample is completely separated and essentially clean of the asphalt/binder.

d) Place a No. 200 (75 µm) sieve on the filter or the extractor and start the vacuum pump.

e) Pour the solvent from the initial rinse onto the No. 200 (75 µm) sieve. If the solvent does not readily pass through the filter, lightly scrape the celite to remove the fines. After the solvent has decanted through the filter, pour approximately 500 mL of solvent into the extractor and let this decant through the filter.

f) Add 200 - 400 mL of solvent to the sample again and decant the solvent into the extractor. Repeat this procedure until the aggregate is clean of asphalt/binder. Normally it will take approximately five rinses to completely clean the sample. (Slag mixtures may require additional rinses.) Rinse the asphalt/binder from the side of the extractor and the sieve with solvent.

g) Allow the vacuum pump to run until all solvent has been decanted through the filter and the filter has a completely dry appearance.

h) If the mixture of water and solvent forms a gel, replace the flask that has been used to collect the solvent, and collect the water rinse separately.

i) Gently stir the layer of celite to break the crust of fines which has formed on the pad. Caution must be exercised to prevent tearing or puncturing the filter paper.

j) Start the pump and pour water through the No. 200 (75 µm) sieve to remove any film left from the solvent.

k) Add enough water to cover the sample and stir well. The water will turn milky-white at this point. After completely stirring, pour the water through the No. 200 (75 µm) sieve, start the vacuum pump, and decant the water into a flask.

l) Repeat 10.2.2 k) until the water is clear. Allow the vacuum pump to run until the filter pad is dry.

m) Rinse the fines accumulated on the No. 200 (75 µm) sieve into the extracted aggregate. Remove the filter ring and lift the filter and place it into another pan. Dry the filter to a constant weight (mass) in the oven or skillet at 221 ± 9 °F (105 ± 5 °C), and weigh immediately upon removal from the oven or skillet.

n) Dry the extracted aggregate to a constant weight (mass) in the oven or skillet at 221 ± 9 °F (105 ± 5 °C), and weigh.

o) The fines in the extracted solvent and water rinse shall be collected in accordance with 10.1.2 g).

10.2.3 CALCULATION WITHOUT FIBERS. The asphalt/binder content in percent is calculated by the following formula:

$$\text{Asphalt/Binder Content, \%} = \frac{W1 - (W2 + W3)}{W1} \times 100$$

Where:

W1 = weight (mass) of sample, g
W2 = weight (mass) of extracted aggregate, g
W3 = weight (mass) of fines in extracted solvent and water rinse, g

10.2.4 USING A VACUUM EXTRACTOR WITH FIBERS.

a) The extraction procedure shall be performed in accordance with 10.2.2.

b) Rinse the fines and fibers accumulated on the No. 200 (75 µm) sieve into the extracted aggregate.

c) Dry the extracted aggregate and fibers to a constant weight (mass) in the oven or skillet at 221 ± 9 °F (105 ± 5 °C), and weigh.

d) Remove the filter ring, lift the filter and place it into a separate bowl. Dry the filter in the oven or skillet at 221 ± 9 °F (105 ± 5 °C), and weigh immediately upon removal from the oven or skillet.

e) Place the extracted aggregate and fibers into the necessary series of sieves. After shaking, the fibers shall be removed from the sieves.

f) Place the fibers and three 1 in. (25 mm) washers into the series of sieves on the No. 4 (4.75 mm) sieve and shake for ten minutes. Remove the fibers from the sieves and weigh separately.

g) The extracted aggregate weight (mass) that is used to calculate the gradation can be determined by subtracting the weight (mass) of the fibers determined in 10.2.4 f) from the combined weight (mass) of extracted aggregate and fibers in 10.2.4 c) and weight (mass) of fines in 10.2.4 d).

h) The fines in the extracted solvent and water rinse shall be collected in accordance with 10.1.2 g).

10.2.5 CALCULATION WITH FIBERS.

a) The asphalt/binder content is calculated by the following formula:

$$\text{Asphalt/Binder Content, \%} = \frac{W1 - (W2 + W3 + W4)}{W1} \times 100$$

Where:

W1 = weight (mass) of test sample, g
W2 = weight (mass) of extracted aggregate, g
W3 = weight (mass) of fines in extracted solvent and water rinse, g
W4 = weight (mass) of fibers, g

b) The fiber content in the mixture is calculated by the following formula:

$$\text{Fiber Content, lbm/t (kg/Mg)} = \frac{W4}{W1} \times 2000\ (1000)$$

10.3 FINES CORRECTION FACTOR.

10.3.1 The extraction procedure shall be performed in accordance with 10.2.2 or 10.2.4.

10.3.2 The fines in the extracted solvent and water rinse shall be collected in accordance with 10.1.2 g).

10.3.3 CALCULATION WITH FINES CORRECTION.

a) A fines correction factor shall be determined by the following formula:

$$\text{Fines Correction Factor(C)} = \frac{W3}{W5}$$

Where:

W3 = weight (mass) of fines in extracted solvent and water rinse, g
W5 = weight (mass) of extracted aggregate passing the No. 200 (75 µm) sieve, g

b) The fines correction factor shall be applied to each subsequent extraction test for each mixture. The correction factor is multiplied by the weight (mass) of extracted aggregate passing the No. 200 (75 µm) sieve, and the calculated weight (mass) is considered the fines in the extracted solvent and water rinse.

c) `The asphalt/binder content in percent is calculated by the following formula:

$$\text{Asphalt/Binder Content, \%} = \frac{W1 - (W2 + (C \times W5)}{W1} X \quad 100$$

Where:

C = fines correction factor
W1 = weight (mass) of sample, g
W2 = weight (mass) of extracted aggregate, g
W5 = weight (mass) of extracted aggregate passing the No. 200 (75 µm) sieve, g

10.4 METHOD C - (USING NO EXTRACTOR).

10.4.1 METHOD SPECIFIC APPARATUS. In addition to the apparatus listed in 5.0 the following apparatus is required for Method C:

a) Pan, round, bowl type, stainless steel

b) No. 200 (75 µm) sieve

c) Suitable containers to collect the extracted solvent and water rinse

10.4.2 USING NO EXTRACTOR.

a) Dry the sample to a constant weight (mass) in accordance with ITM 572.

b) Add enough extraction solvent to cover the sample and stir vigorously. (Soaking the sample after stirring for several minutes may be beneficial in removing the asphalt/binder from the aggregate). Stirring should continue until the sample is completely separated and essentially clean of the asphalt/binder.

c) Place a No. 200 (75 µm) sieve over the container. (Placing the No. 200 (75 µm) sieve in a large funnel prior to collecting the solvent in the container is beneficial).

d) Pour the solvent from the initial rinse through the No. 200 (75 µm) sieve into the container.

e) Add 200 to 400 mL of solvent to the sample and again pour the solvent through the No. 200 (75 µm) sieve into the container. Repeat this procedure until the aggregate is clean of asphalt/binder. Normally it will take approximately five rinses to completely clean the sample. (Slag mixtures may require additional rinses.) Rinse the asphalt/binder from the sieve with the solvent.

f) If the mixture of water and solvent form a gel, replace the container that has been used to collect the solvent, and collect the water rinse separately.

g) Add enough water to cover the sample and stir well. The water will turn milky-white at this point. After completely stirring, pour the water through the No. 200 (75 µm) sieve into the container.

h) Repeat 10.4.2 g) until the water is clear.

i) Rinse the fines accumulated on the No. 200 (75 µm) sieve into the extracted aggregate.

j) Dry the extracted aggregate to a constant weight (mass) in the oven or skillet at 221 ± 9 °F (105 ± 5 °C) and weigh.

k) The fines in the extracted solvent and water rinse shall be collected as in 10.1.2.g). If a high speed centrifuge is used, the extracted solvent and water rinse shall be poured through the centrifuge. The amount of material in the centrifuge cup(s) will be verified to assure that the cup was not overloaded. If the cup was overloaded with fines, then an additional clean cup(s) will be

used, and the extracted solvent and water rinse will be poured through the centrifuge again. This procedure is repeated until the cup is not overloaded.

10.4.3 CALCULATION. The asphalt/binder content in percent is calculated by the following formula:

$$\text{Asphalt/Binder Content, \%} = \frac{W1 - (W2 + W3)}{W1} \times 100$$

Where:

W1 = weight (mass) of sample, g
W2 = weight (mass) of extracted aggregate, g
W3 = weight (mass) of fines in extracted solvent and water rinse, g

11.0 GRADATION. The gradation of the extracted aggregate shall be in accordance with AASHTO T 30 except that decantation through the No. 200 (75 μm) sieve is not required. The entire sample of extracted aggregate is tested for gradation.

12.0 REPORT. The asphalt/binder content and gradation is reported to the nearest 0.1% and the fiber content is reported to the nearest 0.1 lbm/t (0.05 kg/Mg).